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IS 3295-1 (1969): Method of chemical analysis of ferroboron, Part 1: Analysis of carbon, silicon and aluminium [MTD 5: Ferro Alloys]



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Indian Standard
METHODS OF CHEMICAL
ANALYSIS OF FERRO BORON
PART I ANALYSIS FOR CARBON, SILICON
AND ALUMINIUM

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERRO BORON

PART I ANALYSIS FOR CARBON, SILICON AND ALUMINIUM

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERRO BORON

PART I ANALYSIS FOR CARBON, SILICON AND ALUMINIUM

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 30 December 1969, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Ferro boron is used in steel industry as a deoxidizer and as a medium for addition of boron to steel. For this purpose it is necessary that impurities limits and boron content should be correctly determined. In this part, the methods for analysis of carbon, silicon and aluminium have been described. The methods for determination of boron in ferro boron is under investigation and will be covered in Part II.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the ' 1969 Book of ASTM Methods for chemical analysis of metals: Part 32 ', issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes the methods of chemical analysis of carbon, silicon and aluminium in ferro boron in the ranges as specified in IS : 3013-1965†.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down for the purpose of chemical analysis in IS : 1472 (Part I)-1959‡.

*Rules for rounding off numerical values (revised).

†Specification for ferro boron.

‡Methods of sampling ferro-alloys, Part I.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1960*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

4.1 Outline of the Method — The sample is fused with sodium peroxide; silica dehydrated by double evaporation of the acidified extract and determined by hydrofluorization as usual.

4.2 Reagents

4.2.1 Sodium Peroxide — solid.

4.2.2 Dilute Sulphuric Acid — 1 : 1 and 1 : 199 (*v/v*).

4.2.3 Concentrated Sulphuric Acid — sp gr 1.84 (conforming to IS : 266-1961†).

4.2.4 Hydrofluoric Acid — 40 percent (*w/v*).

4.2.5 Potassium Pyrosulphate — solid.

4.3 Procedure

4.3.1 Crush the sample so that it passes through IS Sieve 150 microns. Take 1 g sample and 20 g of sodium peroxide in a 50-ml iron crucible and mix well. Fuse the mixture carefully by revolving the crucible in the outer edge of the flame of a burner at about 700° to 800°C. When the mixture begins to fuse, rotate the crucible so that any unattacked particles attached to the bottom and sides may be attacked. Heat the melt for 10 minutes. Allow the melt to cool to the room temperature, then tap on a solid object to loosen the melt.

4.3.2 Transfer the cold cake to a 500-ml porcelain casserole (having a good glaze) containing 50 ml dilute sulphuric acid (1 : 1). The casserole should be covered. Rinse the crucible with hot water and add the washings to the casserole. Evaporate the solution to dense white fumes.

4.3.3 Continue the fuming for 3 to 5 minutes, cool, add 200 ml of water, and heat to dissolve the sulphates. Filter into a 500-ml casserole using an 11-cm medium paper (to which a little ashless paper pulp has been added), and wash 20 to 25 times with hot dilute sulphuric acid (1 : 199).

*Specification for water, distilled quality (revised).

†Specification for sulphuric acid (revised).

The washing should be thorough to remove all boron. Evaporate the filtrate to dense white fumes, dilute, filter, and wash as previously described. Reserve the filtrate.

4.3.4 Ignite the papers and residue in a platinum crucible first at low temperature to remove all carbonaceous matter and then at 1 050°C for about 15 minutes. Cool and weigh. Moisten the residue with a few drops of water and then add to it 2 drops of concentrated sulphuric acid and 5 to 10 ml of hydrofluoric acid. Evaporate slowly to dryness and finally ignite at 1 050°C for 5 minutes. Cool and weigh.

4.3.5 If any residue remains in the platinum crucible, fuse it with potassium pyrosulphate and dissolve the cold melt in the filtrate reserved under 4.3.3. Reserve the solution for determination of aluminium.

4.3.6 Carry out a blank determination following the same procedure and using the same amount of all reagents, but without the sample.

4.4 Calculation

$$\text{Silicon, percent} = \frac{(A - B) \times 46.72}{C}$$

where

A = weight in g of silica obtained in 4.3.4,

B = weight in g of silica obtained from the blank determination, and

C = weight in g of the sample taken.

5. DETERMINATION OF ALUMINIUM BY THE OXINE METHOD

5.1 Outline of the Method — The silica-free filtrate is treated with methyl alcohol and hydrochloric acid and boron is removed by volatilization as methylborate. Bulk amount of iron is then separated by a single extraction with ether. The residual iron is reduced and complexed with thioglycolic acid. Aluminium is precipitated as aluminium oxinate, filtered, dried and weighed.

5.2 Reagents

5.2.1 Methyl Alcohol

5.2.2 Concentrated Hydrochloric Acid — sp gr 1.16 (conforming to IS: 265-1962*).

*Specification for hydrochloric acid (revised).

5.2.3 Diethyl Ether

5.2.4 Tartaric Acid Solution — 30 percent (*w/v*).

5.2.5 Sodium Hydroxide Solution — 10 percent (*w/v*).

5.2.6 Thioglycolic Acid Solution — Dilute 2 ml of thioglycolic acid (90%) to 100 ml with water. Prepare fresh every 5 days.

5.2.7 Glacial Acetic Acid — 70 percent (*v/v*).

5.2.8 8-Hydroxyquinoline Solution (50 g/l) — Dissolve 50 g of 8-hydroxyquinoline in 120 ml of glacial acetic acid and dilute to one litre.

5.3 Procedure

5.3.1 Evaporate the filtrate reserved under 4.3.5 slowly and reduce the volume to about 100 ml. Adjust the acidity to 6 N with concentrated hydrochloric acid. Add 15 to 20 ml of methyl alcohol and allow the solution to simmer gently. Repeat the addition of methyl alcohol several times until all the boron is removed. Cool and transfer the solution to a 250-ml separatory funnel. Add about 100 ml of ether saturated with concentrated hydrochloric acid and shake for a minute. Discard the non-aqueous layer. Transfer the aqueous layer to a 250-ml beaker and heat on a water-bath to remove ether.

5.3.2 Add 25 to 30 ml of tartaric acid solution and neutralize the solution with sodium hydroxide solution, finally make it alkaline by the addition of 2 to 3 drops more of sodium hydroxide solution. Add few drops of thioglycolic acid solution.

5.3.3 Acidify the solution with glacial acetic acid, heat to 70° to 80°C. Add 25 ml of 8-hydroxyquinoline solution and digest for 30 minutes, without boiling, on a hot-plate. Filter on a tared sintered glass crucible and wash with water. Dry the precipitate in an air-oven at 120° to 130°C for 2 hours. Cool and weigh.

5.4 Calculation

$$\text{Aluminium, percent} = \frac{A \times 5.87}{B}$$

where

A = weight in g of the aluminium oxinate, and

B = weight in g of the sample taken.

6. DETERMINATION OF CARBON BY THE DIRECT COMBUSTION (VOLUMETRIC) METHOD

6.1 Outline of the Method — The sample is burnt in a stream of pure oxygen and the resulting carbon dioxide and excess oxygen after removal

of the contaminants, are collected in a specially jacketed burette and the carbon dioxide absorbed in alkali. On passing the excess oxygen back to the burette, the diminution in volume is read against a scale calibrated directly in percentages of carbon.

6.2 Reagents

6.2.1 Oxygen — 99.5 percent purity free from carbonaceous matter.

6.2.2 Concentrated Sulphuric Acid — see 4.2.3.

6.2.3 Soda Asbestos — Asbestos impregnated with sodium hydroxide in granules of about 2 mm diameter. Avoid contact with air.

6.2.4 Manganese Dioxide — in granules to trap the combustion products of sulphur.

6.2.5 Potassium Hydroxide Solution — containing 300 g of potassium hydroxide per litre.

6.2.6 Dilute Sulphuric Acid Solution — 3 percent (*v/v*).

6.2.7 Methyl Orange — Dissolve 0.05 g of methyl orange in 100 ml of alcohol.

6.2.8 Flux — Lead dioxide, analytical reagent grade..

6.3 Apparatus

6.3.1 The apparatus consists of a source of oxygen and the unit for purifying it, the furnace with the combustion tube; the purification train and the carbon dioxide absorption system. These different parts, which are joined together with connecting tubes forming an air-tight seal, are shown in Fig 1.

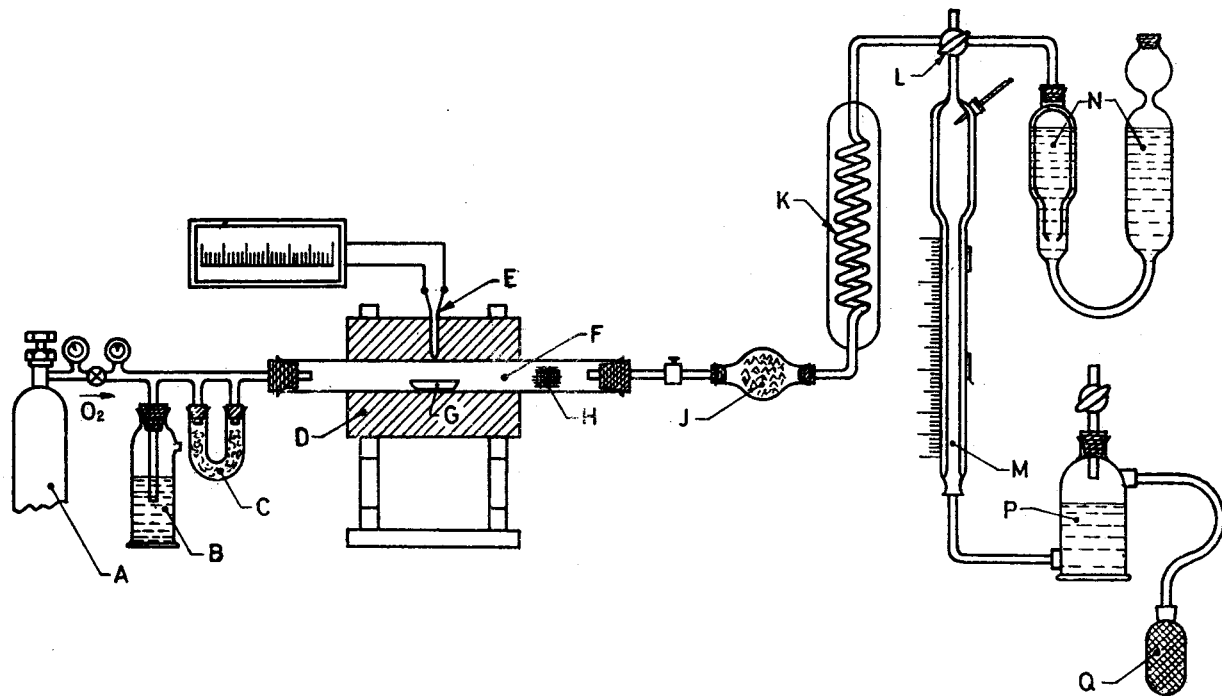
6.4 Procedure

6.4.1 After having verified the pressure tightness of the apparatus, and reaching the temperature 1 000° to 1 100°C in the combustion zone of the furnace, wash the combustion tube with a stream of oxygen, completely fill the special measuring burette *M* and the absorption receiver *N* with the respective liquids manipulating the levelling bottle *P* and tap *L* as appropriate. At this point, tap *L* is closed and the tap on bottle *P* is opened.

6.4.2 Combustion

6.4.2.1 Place 2 g of accurately weighed sample in the calcined porcelain boat. If necessary, add about 2 g of flux (see 6.2.8). Open the combustion tube at the end where the oxygen enters and using a rigid

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A — Source of oxygen (6.2.1) with pressure regulating valve.

B — Mercury valve.

C — Unit for drying and purifying the oxygen, containing concentrated sulphuric acid (6.2.2) and soda asbestos (6.2.3) separated by glass wool (diameter of tubes 25 mm and height 100 mm, approximately) connected by tubing.

D — Wire wound or resistor rod furnace, made of carborundum or metal and capable of a temperature for combustion up to 1200°C .

- E* — Thermocouple for measuring the temperature. The tip of the thermocouple, protected by a sheath, is placed near the external surface of the combustion tube. The relation between the internal tube temperature and the pyrometer reading should be established.
- F* — Combustion tube made of refractory material which is not porous at the test temperature, internal diameter 20 to 30 mm and at least 650 mm long so that the ends of the tube remain cold during combustion.
- G* — Boat made of refractory material (length 80 to 100 mm, width 12 to 14 mm and depth 8 to 9 mm), calcined in a stream of oxygen at the temperature of use.
- H* — Plug of calcined asbestos wool or quartz wool to filter the gas.
- J* — Glass bulb, filled with manganese dioxide granules (6.2.4) and quartz wool, to trap any sulphur combustion products and dust carried over by the stream of gas.
- K* — Capillary coil, water cooled (inside diameter 1.5 mm).
- L* — Tap used for connecting the coil *K* with the special calibrated burette *M*, or with the absorption receiver *N*, or with the atmosphere.
- M* — Special calibrated burette, cooled by a water jacket, with an enlarged portion at the top fitted with a thermometer capable of readings correct to 0.05°C. The capacity of the burette should be about 400 ml for carbon contents up to 1.5 percent. The bottom of the burette is connected by a rubber tube to a levelling bottle *P*, on the side of the calibrated lower portion of the burette there is a scale capable of sliding along the burette itself.
- N* — Absorption receiver with liquid trap, containing a solution of potassium hydroxide solution (6.2.5) used for absorbing the carbon dioxide.
- P* — Levelling bottle, containing water, dilute sulphuric acid solution (6.2.6), coloured red with methyl orange (6.2.7) with tap and compression valve *Q*.
- Q* — Rubber compression valve.

FIG. 1 COMBUSTION APPARATUS FOR DETERMINATION OF CARBON BY VOLUMETRIC METHOD

nickel wire, place the boat and the sample in the middle of the heating zone of the tube. Quickly close the tube and after about half a minute, during which time the oxygen should be turned off, connect the tube with the measurement burette *M* by means of tap *L* and at the same time pass a stream of oxygen through the apparatus (liquid in the burette should descend slowly).

6.4.2.2 The stream of oxygen should be regulated so that at the end of combustion there is enough space in the measurement burette to receive the carbon dioxide formed and also the excess oxygen required for completely removing the last traces of carbon dioxide from the combustion tube *F*, from the bulb *J*, and from the capillary coil *K*. When the level of the liquid in the burette *H* continuing its descent, comes near to zero of the scale turn the tap *L* which cuts off communication of burette *M* with the combustion tube *F*, stop the stream of oxygen, and remove the boat from the combustion tube. It is necessary to find out whether combustion has been duly effective, by examining the fused mass in the boat removed from the furnace.

6.4.3 Determination

6.4.3.1 After two minutes, the measuring liquid in the burette and in the measuring bottle *P* with the tap still open, should be brought to the same level and the graduated scale should be moved so as to bring the zero to the level of the liquid.

6.4.3.2 Then, by means of tap *L*, connect burette *M* with the absorption receiver *N*, close the tap on the levelling bottle *P*, and by means of the compression valve *Q*, slowly pass the gas into the absorption receiver *N*.

6.4.3.3 Open the tap on the levelling bottle *P*, lower the latter, and bring back the gas into the measurement burette *M*.

6.4.3.4 Recommence passing the gas into the potassium hydroxide solution in absorption receiver *N*, bring back all the gas into the measurement burette, close the stopcock end and, after obtaining the same level of the liquids, read on the scale the percentage carbon and the temperature of the gas correct to $\pm 0.05^{\circ}\text{C}$.

6.4.3.5 The burette is graduated to measure directly in percentage up to 4.5 percent carbon on the basis of 1 g sample taken. From the reading on the scale the percentage of carbon could be calculated on the basis of 2 g sample taken in **6.4.2.1**. The percentage read should be corrected for temperature and pressure recorded at the time of determination by means of a correction factor.

6.4.4 Blank Test — Blank test should be carried out by combustion of a weight of metal of known carbon content in the presence of amounts of the normally used fluxes carefully weighed.

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